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Synthesis of superhard phases of boron nitride in a rotational diamond anvil cell

V. Levitas, J. Hashemi, W. Mathis, M.Holtz, and Y. Ma, Center for Mechanochemistry and Synthesis of New Materials, Texas Tech University

Beamline(s): X17C, X17B

Introduction: It is well known from numerous experiments that superposition of plastic shear on hydrostatic pressure in a Rotational Diamond Anvil Cell (RDAC) leads to a number of new phenomena including i) the reduction of the phase transformation (PT) pressure, and ii) formation of new phases that would not be produced without rotation. The PT behavior of the boron nitride (BN) system in a RDAC has not been investigated. Superhard phases of BN, wurtzitic wBN and especially cubic cBN, have important technological applications due to their hardness, second only to diamond, and their superior chemical and thermal stability. In industry, superhard phases of BN are generally synthesized under pressures greater than 4 GPa and temperatures exceeding 1600 K. Synthesis of cBN and wBN at room temperature could be of great fundamental and technological importance. In this paper, we report the strain-induced synthesis of a mixture of cBN and wBN at room temperature and high pressure in RDAC.

Methods and Materials: At the Center for Mechanochemistry and Synthesis of New Materials at Texas Tech University, we possess a RDAC with a culet diameter of 0.5 mm (capable of 80 GPa pressure), Figure 1. The RDAC was manufactured for the center by the Institute for Superhard Materials (Kiev, Ukraine). The RDAC system is equipped with sensors to monitor i) the thickness of the sample (with +/- 1 micron accuracy), ii) the force exerted on the anvil (up to 10 KN), and relative alignment of the anvils (+/- 1 degree). A detailed description of a similar RDAC and the corresponding measurement techniques, and some experimental results can be found in our joint paper [1]. A stainless steel gasket with a 250 μm diameter hole was packed with hexagonal BN. The initial thickness of the sample was measured at 212 μm. The gasket was placed in the RDAC, Figure 1, and pressure was applied to the sample to 5 GPa.

The hole containing the hBN was observed to grow during the pressurization process. Pressure was increased to 10 GPa at which point the thickness of the sample was measured to be 58 μm. At this point, plastic shear was induced into the sample by rotating the diamond anvil by approximately 120^o (60^o CCW followed by 60^o CW). The thickness was dropped to approximately 24 μm. No discernable additional enlargement of the hole was observed at this point. Pressure was then increased to 25 GPa with a measured sample thickness of 11 μm. The sample was subjected to plastic shear by rotating the anvil in both directions for a total amount of 240^o. The thickness of the sample reduced to 5 μm after this rotation stage.

Results: The sample was removed from the RDAC and observed under a light microscope. There was a noticeable change in the color of the sample from white to dark brown. Raman Scattering was performed on the compressed sample. The Raman spectra of the compressed sample showed broadened peaks corresponding to a mixture of residual hBN, cBN, and wBN, Figure 2. Synchrotron radiation tests performed in Brookhaven National Lab. also showed that the material synthesized is a mixture of superhard cBN and wBN phases, in good agreement with our Raman results. We cannot prove that the superhard phase appeared at the first rotation at 10 GPa, but there are some indirect evidences that this was the case. This is the first synthesis of superhard BN phases in RDAC at room temperature.

After completion of the loading process and upon close examination of the surface of the anvil, deep scratches with circular patterns were observed. The scratches are indicative of the high hardness of the synthesized sample. Additionally, particles of sizes up to a few microns were observed to have bonded to the surface. These particles could not be removed by any mechanical means, Figure 3. Both Raman and Synchrotron X-ray techniques yielded inconclusive results on the nature of these particles. Research is continuing to determine the make-up and structure of these particles. We have not ruled out the possibility of a new phase belonging to the B-C-N system.

Conclusions: Complete PT from hBN to a mixture of superhard cBN and wBN phases was observed at room temperature, a pressure level between 10 and 25 GPa, and extensive plastic shear in a RDAC. Also, the very strong bonding of hard particles of unidentified structure on the surface of the anvil is of great interest requiring further research.

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References: Novikov, N. V., Polotnyak, S. B., Shvedov L. K. and Levitas, V. I. (1999). Phase Transitions Under Compression and Shear in Diamond Anvils: Experiment and Theory. J. of Superhard Materials, **3**, 39-51.

Figure 1. The Rotational Diamond Anvil Cell (RDAC).

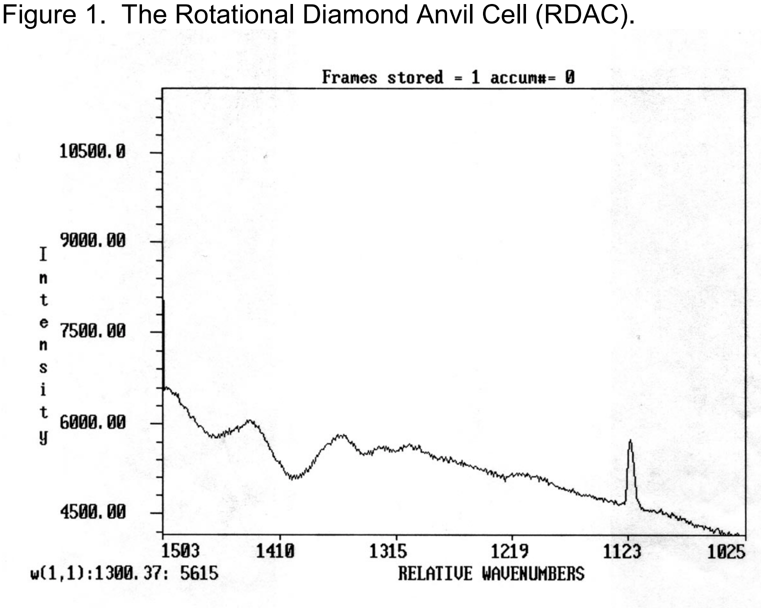

Figure 2. The Raman spectra of the synthesized sample**.**

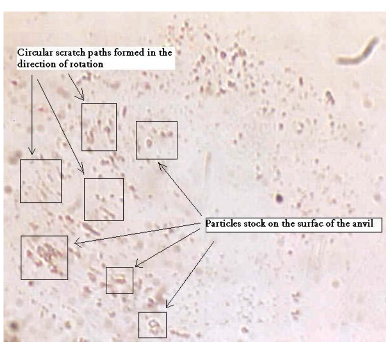

Figure 3. A photomicrograph showing the surface of the damaged anvil.